Receipt date: 09/07/2004 WO 2004/005183

10

15

10506981 - GAU: 1793 PCT/US2003/003393

OK TO ENTER

/JF/

07/31/2008

## WE CLAIM:

- 1. A method for preparing nanoparticles comprising cerium oxide and zirconium, wherein the method comprises the steps of:
- 5 (a) providing a first aqueous solution comprising zirconium oxychloride;
  - (b) mixing the first aqueous solution with a second aqueous solution comprising a first component selected from one of cerium nitrate and hexamethylenetetramine to form a first mixture;
  - (c) mixing the first mixture with a third aqueous solution comprising a second component selected from cerium nitrate and hexamethylenetetramine to form a second mixture, wherein the second component is different from the first component;
    - (d) maintaining the second mixture at a temperature no higher than about 320 °K to form nanoparticles therein;
    - (e) separating the nanoparticles formed in step (d) from the second mixture; and
      - (f) sintering the nanoparticles separated in step (e) in air at a temperature ranging between about 500° C to about 1100°C.
- 20 2. The method of Claim 1, wherein the first aqueous solution has a concentration of zirconium oxychloride ranging from about 0.005 M to about 0.1 M.
  - 3. The method of Claim 1, wherein the second aqueous solution comprises cerium nitrate in a concentration ranging from about 0.005 M to about 0.1 M.
- 4. The method of Claim 1, wherein the third aqueous solution comprises cerium nitrate in a concentration ranging from about 0.005 M to about 0.1 M.
  - 5. The method of Claim 1, wherein the second aqueous solution comprises hexamethylenetetramine in a concentration ranging from about 0.01 M to about 1.5 M.
- 6. The method of Claim 1, wherein the third aqueous solution comprises hexamethylenetetramine in a concentration ranging from about 0.01 M to about 1.5 M.

Receipt date: 09/07/2004 10506981 - GAU: 1793 **WO 2004/005183** 10506981 - GAU: 1793

7. The method of Claim 5, wherein the second aqueous solution comprises hexamethylenetetramine in a concentration of hexamethylenetetramine ranging from about 0.5 M to about 1.5 M.

8. The method of Claim 6, wherein the third aqueous solution comprises hexamethylenetetramine in a concentration of hexamethylenetetramine ranging from about 0.5 M to about 1.5 M.

5

15

25

- 9. The method of Claim 1, wherein step (d) comprises stirring the second mixture while it is being maintained at a temperature no higher than about 320°K to form nanoparticles therein.
- 10 10. The method of Claim 1, wherein the first and second mixtures are formed in a container having a mechanical stirrer, and the first mixture and the third aqueous solution are mixed with the mechanical stirrer to form the second mixture.
  - 11. The method of Claim 1, wherein step (e) comprises maintaining the second mixture at a temperature no higher than about 320 °K for a time period between about 2 hours and about 24 hours.
  - 12. The method of Claim 11, wherein the time period is between about 5 hours and about 24 hours.
  - 13. The method of Claim 12, wherein the time period is between about 12 hours and about 24 hours.
- 20 14. The method of Claim 1, wherein step (e) comprises centrifuging the second mixture to separate the nanoparticles from the second mixture.
  - 15. The method of Claim 1, wherein the second mixture is formed in a container, and the method comprises positioning the container inside a centrifuge and centrifuging the second mixture after formation of the nanoparticles therein for separating the nanoparticles from the second mixture.
  - 16. The method of Claim 1, wherein the nanoparticles separated in step (e) are at least in part crystalline.
  - 17. The method of Claim 1, wherein after step (f) is performed, the nanoparticles are at least in part crystalline.
- 30 18. The method of Claim 1, wherein the sintering of the nanoparticles in step (f) takes place at a temperature of about 550 °C.

Receipt date: 09/07/2004 10506981 - GAU: 1793 WO 2004/005183 PCT/US2003/003393

19. The method of Claim 1, wherein the sintering of the nanoparticles in step (f) takes place at a temperature of about 900 °C.

20. The method of Claim 1, wherein the molar percentage of zirconium in the nanoparticles comprising cerium oxide and zirconium is in the range of about 20% to about 75%.

5

10

15

20

25

- 21. The method of Claim 1, wherein the first aqueous solution is provided in a container, and the second and third aqueous solutions are mixed with the first solution and the first mixture, respectively, by pumping the second and third aqueous solutions into the container through a plurality of inlets which are distributed throughout the container.
- 22. A method for preparing nanoparticles comprising cerium oxide and zirconium, wherein the method comprises the steps of:
  - (a) providing a first aqueous solution comprising a first component selected from one of cerium nitrate and hexamethylenetetramine;
  - (b) mixing the first aqueous solution with a second aqueous solution comprising a second component selected from one of cerium nitrate and hexamethylenetetramine to form a first mixture, wherein the second component is different from the first component;
  - (c) maintaining the first mixture at a temperature no higher than about 320 °K for about 1 to about 5 hours;
  - (d) after step (c) mixing the first mixture with a third aqueous solution comprising zirconium oxychloride to form a second mixture;
  - (e) maintaining the second mixture at a temperature no higher than about 320 °K to form nanoparticles therein;
  - (f) separating the nanoparticles formed in step (e) from the second mixture; and
  - (g) sintering the nanoparticles separated in step (f) in air at a temperature in the range of about 500° C to about 1100°C.
- A method for preparing nanoparticles comprising cerium oxide and zirconium,
  wherein the method comprises the steps of:

Receipt date: 09/07/2004

5

10

10506981 - GAU: 1793 PCT/US2003/003393

- (a) providing a first aqueous solution comprising zirconium oxychloride;
- (b) mixing the first aqueous solution with a second aqueous solution comprising hexamethylenetetramine to form a first mixture, wherein the first and second aqueous solution are mixed for an amount of time sufficient to allow formation of a precipitate in the first mixture;
- (c) mixing the first mixture with a third aqueous solution comprising a cerium nitrate to form a second mixture;
- (d) maintaining the second mixture at a temperature no higher than about 320 °K to form nanoparticles therein;
- (e) separating the nanoparticles formed in step (d) from the second mixture; and
  - (f) sintering the nanoparticles separated in step (e) in air at a temperature ranging between about 500° C to about 1100°C.
- 15 24. The method of Claim 23, wherein the first and second aqueous solution are mixed in step (b) for between about 1 hour to about 2 hours.